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DEVELOPMENT OF SPECIALIZED EQUIPMENT TO MEASURE PIEZOELECTRIC C--ETC(U)
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(6) Development of Specialized
Equipment to Measure Piezoelectric Coefficients
of Polymer Piezoelectric Materials

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1.0 Abstract

Specialized equipment has been developed to measure piezoelectric g and d coefficients of polymer piezoelectric materials. It is likely that the same equipment will allow the evaluation of these properties for composite flexible lead titanate zirconates as well. However; samples of these were not made available and thus not tested. By means of a special adaptor, the same equipment may be used as a standard Berlincourt d_{33} Meter Channel Prod. Model Number CPDT 3300.

Because of the nature of the polymer film there are several limitations in the use of the equipment described to measure their properties. These include both a restricted frequency range and test sample size. Accuracy of the equipment is limited to approximately $\pm$ 10% for d_{33} and $\pm$ 15% for the measurement of the transverse coefficients.

2.0 Introduction

The electromechanical test set-up developed for the evaluation of the polymer films is based on the Berlincourt Piezo d_{33} Meter Channel Products Model CPDT 3300 (description attached). The basic difference is in the manner of supporting the film which calls for the use of special adaptors. These are illustrated in figure 1.

Reference to figure 1 shows as extension srm attached to the upper brace which includes thumbscrews for clamping the two ends of a strip of stretched polymer film. The lower probe includes a thin support member around which the film is looped.

The film is dynamically driven along its length while under a slight mechanical bias. Charge is generated on the length-width face and this allows measurement of d_{31}. The piezoelectric g_{31} coefficient can also be determined by the same method of loading. For this the switch in the special insert between the Force head test output and the diplay unit should be placed in the g position. This provides a capacitance in series with the test piece which overrides the effect of its own capacitance. With its value properly chosen a direct value of g_{31} is provided on the digital display. The mathematical treatment of the measurement of both d_{31} and g_{31} is developed later in this report.

In addition to these features the equipment was designed to be used as a standard d_{33} Meter as well by means of an additional special upper probe illustrated in figure 1(c). This is to be attached to the upper brace. The lower support used for the polymer film is removed and any of the other standard probes may be inserted. The equipment is then ready for use as a standard d_{33} Meter. In this mode therefore d_{33} of the film may also be measured. However; because of the low value involved, the procedure outlined in one of the attachments* should be followed.

* "Step by Step Instructions on Use of Channel Products CPDT 3300 to Measure d_{33} values below 100×10^{-16} c/N and above 5×10^{-12} c/N."

Despite the apparent simplicity of the final solution much difficulty was encountered with its early evaluation and considerabledebugging was effected. Attempts to obtain results that were vertually independent of frequency, mechanical bias and length of sample proved difficult and as a result some limitations are present especially for measurements of transverse mode coefficients.

A frequency dependency exists which is a result of a low system resonance inherent because of the high compliance of the test load. Thus not only should the operating frequency be limited to a range below 100 Hz, test samples less than 2.5 cm - support length by approximately 6mm or more in width are recommended. This is especially so with films of 25 or 30 micron thickness which were used in tests to be discussed later. It is likely that with thicker pieces, longer and/or narrower test samples may be evaluated. Further experimental evaluations are recommended to ascertain the best operating conditions in such cases.

Tension is somewhat critical for all measurements -33 as well as 31 (32) and only slight or gentle bias is recommended. Care should be taken to eliminate sagging and wrinkles. A force gauge mounted on the Force head provides a quantitative guide to setting tension.

For measurements of d_{33} only test samples of small area should be used. A convenient size was found to be squares of approximately 5mm x 5mm. In this case, probe C (see Operating Manual CPDT 3300) should be placed in the lower position.

3.0 Discussion

3.1 Flexure Approach

An approach contemplated early and described in the Channel Products proposal dated November 2, 1977 was found to be ineffective. In addition it appeared to lack flexibility of utilization and thus was discarded early.

The concept alluded to involved loading the polymer film in a bending mode by attaching it to a substrate of geometry and stiffness to be unaffected by the film. This appeared at first to be a reasonable approach since it lends itself to a simple analytical solution. For example: the stress developed in the film for a concentrated force at the center of a span for the simply supported flexure member is the maximum fiber stress determined from the theory of flexure. The output voltage and/or charge are related to the average stress developed and appropriate piezo coefficients and geometry. Hence both pertinent coefficients would be easily obtained.

However, the practical adaptation of this concept proved difficult. It was not possible to attach the film to the substrate with 100% mechanical coupling unless destructive techniques were to be used, namely bonding and the like. This would not allow for flexibility of measurement whereby the sample could then be used in other modes or for adapation to other transducer designs. Attaching the film by any other technique failed to yield good results. Values as low as 10% of true level of g and d coefficients were obtained.

3.2 Length Expander Approach

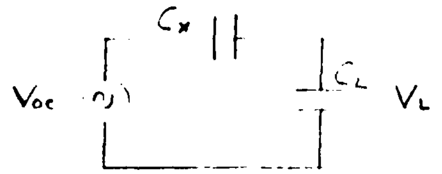
It was apparent therefore that other techniques would be necessary. The next most obvious choice was to clamp both ends of a strip of convenient size and apply the dynamic force transversely, (i.e. parallel to length and perpendicular to the polar axis) while maintaining gentle tension; the method that evolved is illustrated in figure 1(a) and was discussed earlier. A second form is shown in figure 1(b).

In the preferred case (figure 1(a) the film is placed under tension with a force tending to pull the force head diaphragm in an upward direction. Conversely in figure 1(b) is shown the opposite situation.

The method of attachment of the film is the same in both cases. One end of the sample is clamped between the center insulator and the silvered contact by means of the thumbscrew. The film is then looped under (over in the second case) the center member of the lower probes. The other end is then clamped as above with the opposite thumbscrew. In either case very gentle tension is to be maintained so as to avoid sagging and/or wrinkles.

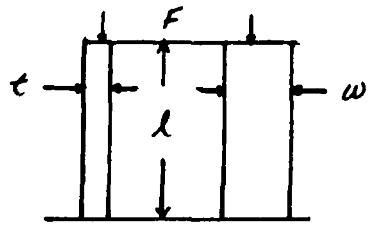
3.3 Mathematical treatment to determine required capacitance load for a direct reading of d_{31}

A review of the proceedure used to set-up the required value of C_L for the digitally displayed value of d_{31} or d_{32} is outlined below. The equivalent circuit also is shown below:



1. Set up expression for V_{oc} open circuit voltage
2. " " " " C_x capacitance of test element
3. " " " " V_L voltage across C_L load
4. Combine (1) and (2) into (3)
5. Set up expression for V std. (Internal calibration element in force head)
6. Form the ratio of V_L/V std.
7. The product of (6) and "d_{33}" of the standard is the reading "R" digitally displayed
8. Solve for "R"
9. Set d_x - unknown d constant - R
10. Solve for C_L to read d_x in appropriate units

Example:



1. $V_{oc} = (g_{31}/w)F$ (by definition $g_{31} = (V_{oc}/t)(F/wt)$ = field/stress

2. $C_x = K_{11}^T \epsilon_o \ell w/t$ (parallel plate capacitance formula)

3. $V_L = V_{oc}\, C_x/C_L$ for $C_L >> C_x$

4. $V_L = (g_{31}/w)F \quad (K_3^t \epsilon_o \ell w/t)(1/C_L)$

$= g_{31}\, K_3^T\, \epsilon_o \ell/t\, (F/C_L)$

$= d_{31}\, (\ell/t)(F/C_L)$ (since $d_{31} = K_3^T\, \epsilon_o g_{31}$)

5. $V_{std} = "d_{33}"/C_{std}\, F$

6. $V_L/V_{std} = [(d_{31} \ell/t)(1/C_L)]/(d_{33}/C_{std})$

7. $R = (V_L/V_{std})\, d_{33} = (d_{31} \ell/t)(C_{std}/C_L)$

8. R = in 10^{-12} units

9. For d_{31} in 10^{-12} units

$d_{31}/R = t/\ell \quad (C_L/C_{std}) = 1$

or $C_L = (\ell/t)(C_{std})$

$C_{std} = 10^{-6}$ F for the CPI d_{33} Meter

Thus to obtain a direct readout of d_{31} for a part 2.5 cm long x 0.25 cm thick:

$$C_L = \text{should be } \frac{2.5}{.25} \times 10^{-6}\ F$$
$$= 10\,\mu F$$

With the polymer film, thicknesses are usually considerably less. For example; the samples available were 28.5 μm thick,

Thus for ℓ 2.54 cm $\ell/t = 891\,\mu F$

By reducing this to 89.1 μF the display reading would have to be reduced by a factor of 10. Thus a value shown as 175 would in fact indicate a d_{31} of 17.5×10^{-12} c/N.

In order to illustrate the use of the above procedure we could have calculated C_L with this in mind - that is let reading be in 10^{-11} units instead. Refer to Step (9).

Thus $d_{31}/R = (10^{-11}/10^{-12})(C_L/C_{std})\ t/\ell = 1$

Indicating display value to be divided by 10 i.e.,

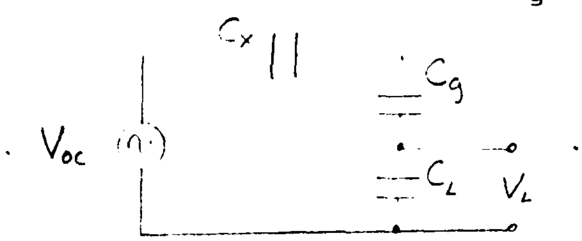
$$C_L = 10^{-1}\ (\ell/t)\ C_{std}$$

or

$$C_L = 89.1\,\mu F$$

3.4 Mathematical treatment to determine required capacitance load for direct reading of g_{31}.

In this case a small series capacitance C_g is added as shown below:



C_g is inserted in order to reduce if not completely eliminate the effect of C_x element (film) capacitance.

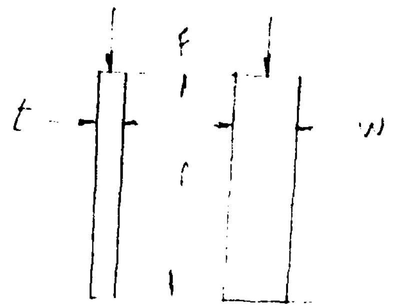
That is $\quad C_g \ll C_x$

also $\quad C_g \ll C_L$

Again in outline form:

1. Set up expression for V_{oc} - *open circuit voltage*
2. " " " " V_L
3. Combine steps (1) and (2)
4. Set up expression for V_{std} - internal calibration standard in force head
5. Form the ratio V_L/V_{std}
6. The product of (5) and "d_{33}" at the standard is the reading "R" digitally displayed
7. Solve for "R"
8. Set g_x - unknown constant = R
9. Solve for ratio C_g/C_L to read g_x in appropriate units

Example:



1. $V_{oc} = (g_{31}/w)F$
2. $V_L = (C_g/C_L)V_{oc}$
3. $V_L = (C_g/C_L)(g_{31}/w)F$
4. $V_{std} = "d_{33}"/C_{std}\ F$
5. $V_L/V_{std} = (C_g/C_L)(g_{31}/w)(C_{std}/"d_{33}")$
6. $R\binom{\text{in } 10^{-12}}{\text{units}} = (V_L/V_{std})\ "d_{33}" = (C_g/C_L)(g_{31}/w)\ C_{std}$
7. For R in 10^{-3} units
8. $g_{31}/R = 10^{-3}/10^{-12} = w(C_L/C_g)(1/C_{std})(10^{-3}/10^{-12})$
9. $C_g/C_L = 10^{-9}\ w/C_{std}$

$C_{std} = 10^{-6}$ F

$C_g = 20 \times 10^{-12}$ F, this generally satisfies conditions indicated earlier ($C_g \ll C_x$ and $C_g \ll C_L$)

for width = .25" = 6.35 mm then;

$$C_L \quad \frac{20 \times 10^{-12} \times 10^{-6} \times 10^{9}}{.635 \times 10^{-2}}$$

$$= 2/.635\,\mu F = 3.15\,\mu F$$

Since we have a double loop of film, the value for w should be twice that indicated or:

$$C_L = 1.57 \mu F$$

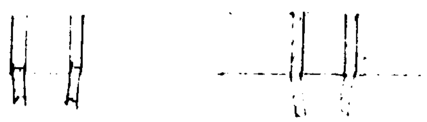
and

$$C_g = 20 \text{ pF}$$

for direct reading of g_{31} in 10^{-3} units.

3.5 Special Note applying to the measurement of g_{31}

The portion of film within the clamped regions represents dead or unstressed capacitance and thus serves to act as an additional shunt load. To minimize or eliminate this effect this portion should be reduced to a minimal amount, that is clamping should be effected over the smallest amount of material possible. This is illustrated below:



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4.0 Test Results

4.1 Preliminary Tests to Determine d_{31} and g_{31} of PVF_2

Two tests were used to determine the piezoelectric transverse coefficients of d and g of PVF_2 samples submitted for this program. One was a static test and the other dynamic.

In the static test, use was made of the upper brace and support systems developed and discribed earlier. The brace was removed from the Force head and reattached 180° from its standard position. This allows sufficient room when placing the head on a platform or at the edge of the work table to attach a Chatillon 0 1000 gram gauge. The set up is illustrated in figure 2 .

In a preferred technique, a bias load (approx. 150 grams) provided by the weight of the gauge itself is first applied. The output voltage generated by the film is monitored by an electrometer or other high impedance detector. The input to the detector is kept shorted to establish an equilibrium with the initial bias. This is removed simultaneous to the application of a superimposed test load to acheive an output voltage of some convenient level.

The piezoelectric g coefficient is found as follows:

$$g_{31} = \left(V/F\right) 2\,w$$

V = open circuit output votage, in volts

F = average load (in newtons) of several trials

w = width of test piece in meters

In addition, measurement of capacitance and subsequent calculation of dielectric constant allows for a determination of d_{31}.

Results of such tests yielded the following:

Sample A $g_{31} = 155 \times 10^{-3}$ V m/N

$K_3^T = 14.5$

$d_{31} = 19.9 \times 10^{-12}$ c/N

Assume; $Y_{11}^E = 3.6 \times 10^9$ N/m^2

$k_{31} = 0.105$

Sample B $g_{31} = 155 \times 10^{-3}$ V m/N

$K_3^T = 13.0$

$d_{31} = 17.8 \times 10^{-12}$ c/N

Again Assume; $Y_{11}^E = 3.6 \times 10^9$ N/m^2

$k_{31} = 0.100$

As a verification of the values of coupling obtained with the aid of static tests, a dynamic measurement of k_{31} was also made. This was effected by utilizing a piece of stretched film as a 3 terminal device - as a piezo transformer - as shown in figure 5.

A simple non rigorous solution to this arrangement - using an electromechanical equivalent circuit approach yields the following:

$$V_{out} = \left\{\left[(g_{31}/w) \Big/ \left[(1/Y_{11}^E)(\ell_2/wt)\right]\right\}\left(\tfrac{1}{2} d_{31}\right)(\ell_1/t)V_{in}$$

which reduces to $k_{31}^2 = 2\, V_{out}/V_{in}$ for frequencies well below resonance.

This expression differs from the one derived by J.G. Linvill* which follows:

$$k_{31}^2 = 2\, V_{out}/(V_{out} + V_{in})$$

*PVF_2 - Models, Measurements, Device Ideas by J.G. Linville- Technical Report No. 4834-3 NSF GRant Eng.75-22329 Stanford University March 1978.

However for low values of k_{31} ($V_{in} >> V_{out}$) as is the case for polymers - the two expressions are equivalent and either may be used. Results of this test yeilded:

Sample A k_{31} = 0.104

Sample B k_{31} = 0.101

These are in good agreement with the calculated data shown above.

4.2 d-Meter Measurements

Data to be presented here were taken on material represented by sample B since more of it was made available for test purposes.

4.2.1 Reveiw of d-meter set up

Among the attachments included with this report are two that are pertinent. One was referred to earlier; namely "Use of Channel Products CPDT 3300 with the 110μF Decade Capacitance Box" and the second "Technical Note No. 1 - Theory of the use of Channel Products CPDT 3300 d_{33} meter to measure d_{31} directly on Piezoelectric Ceramic Tubes and Bars". In addition reference should be made to the Operating Manual.

Basically the following steps should be used:

For polymer films - the d_{33}/d_{31} switch on the rear panel should be in the d_{31} position. This removes the 1μF load normally in place for standard 33 measurements of piezo ceramics and places the Decade capacitance box provided (and appropriately connected to the BNC connector at the rear of the instrument) in parallel with the test sample. The d_{33}/Force switch should be in the d_{33} position.

The value of C_L should be determined as in sections 3.3 and 3.4.

In addition the switch located in a special box connected to the test output at the force head should be placed in accordance with the desired parameter.

In the g position, a small capacitance (20 pf) is placed in series with the test element as discussed in section 3.4.

4.2.2 Anisotropy of d and g of PVF_2

In one series of tests the anisotropy of d and g coefficients were determined on the uniaxially stretched PVF_2. Data were taken with the d-meter (using both types of lower probes) on samples each approximately 5 cm by 6.3 mm by 28.5 microns. The effective or clamped length was approximately 2.5 cm or half of the 5 cm total length. Load capacitance for these measurements were determined in accordance with sections 3.3 and 3.4.

Curves of d_θ and g_θ as a function of θ angle of stretch - are shown in figure 4. The values of d_θ seem in agreement at least for $\theta = 0^\circ$ ($d_\theta = 18.5 \times 10^{-12}$ c/N) which is in good agreement with the value shown earlier (Sample B - 17.8×10^{-12} c/N). However; g_θ at $\theta = 0^\circ$ is a bit low. Part of this is due to the shunting effect of the unstressed capacitance (film within clamped region) but this only accounts for perhaps 5%.

It is also likely that these materials are not completely homogeneous and thus the section used for these measurements are in fact different than the piece tested earlier.

At any rate the agreement is reasonably good.

4.2.3 Influence of length of test sample

Another series of tests were performed to show the influence of length of test sample on d_{31} and g_{31}.

These data are shown in figure 5 wherein both d_{31} and g_{31} are plotted as a function of clamped length. Again both types of lower probes were used.

It is believed that the lower values of both d and g at greater lengths are a result of loss of force (analogous to voltage drop within a generator of moderate internal impedance). Thus as the compliance of the load increases - as with increasing length (and/or decreasing cross sectional area), a greater velocity causes an increase in force drop. The calibration standard monitors in essence the open circuit force rather than force at the load. Hence relativly stiff samples of the film are recommended for test purposes.

4.2.4 d_{33} Measurements of PVF_2

This is a routine measurement with the d-g switch placed in the d position and the d_{33}/d_{31} switch placed in the d_{31} position. As explained earlier this removes the standard 1μF load across the test element. This allows for a variable load (Decade Capacitance Box DC-1) to be connected instead. This increases the resolution of measurement for d_{33} values of less than 100. In this case C_L was set to 0.1μF and readings of d_{33} of several test pieces were displayed as:

d_{33} 173 x 10^{-11} c/N

or

d_{33} 17.3 x 10^{-12} c/N

The actual range of a number of samples was from 16.0 through 18.5 x 10^{-12} c/N.

5.0 Conclusions

Despite the limitations in the measurement of d and g coefficients of polymer film, the d-meter described should be a useful tool. Relative measurements of various samples and/or materials are feasible.

In addition, direct measurement of d_{33} is more reliable especially with the aid of the external variable load capacaitance provided to increase resolution.

Finally, the equipment is also useful as a standard d_{33} meter with additional features such as a d_{33}/d_{31} switch and variable load

to accommodate a variety of piezo ceramics as well as provision for g constant measurements.

It is conceivable that improvements can be made in the accuracy by appropriate modifications. Two of these include a) more compliant diaphragm to reduce the influence of load compliance and b) a relocation of the force sensing standard to more precisely indicate load force. A more compliant diaphragm would also require other internal modifications to keep the system resonance high.

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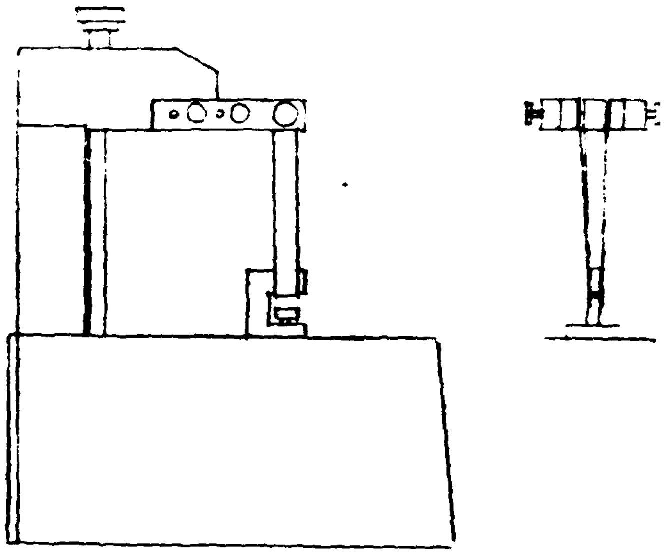
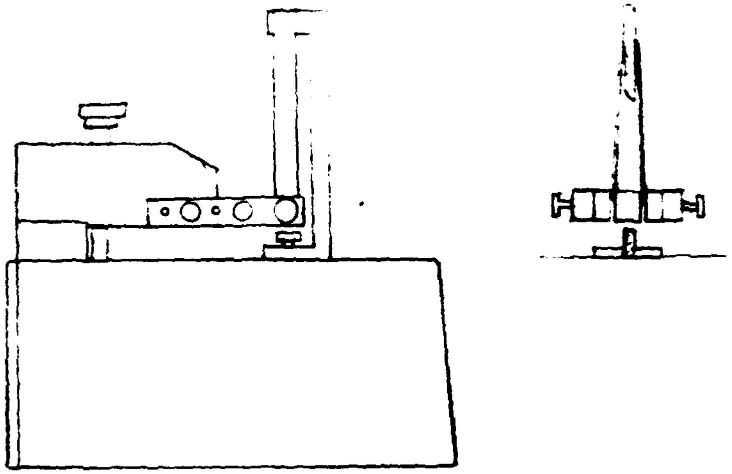
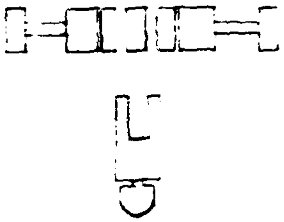


a) WITH LOWER PROBE E



b) WITH LOWER PROBE F



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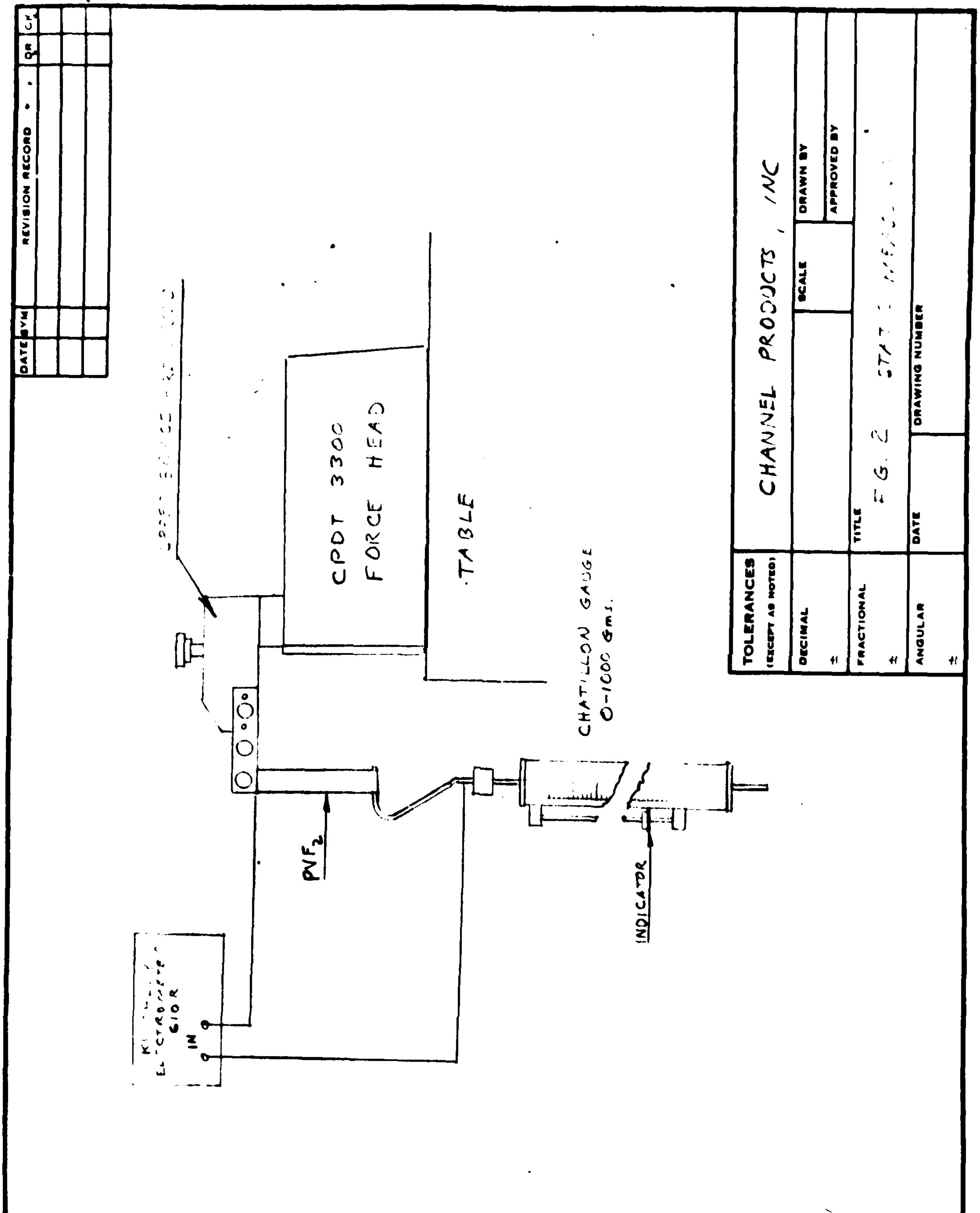
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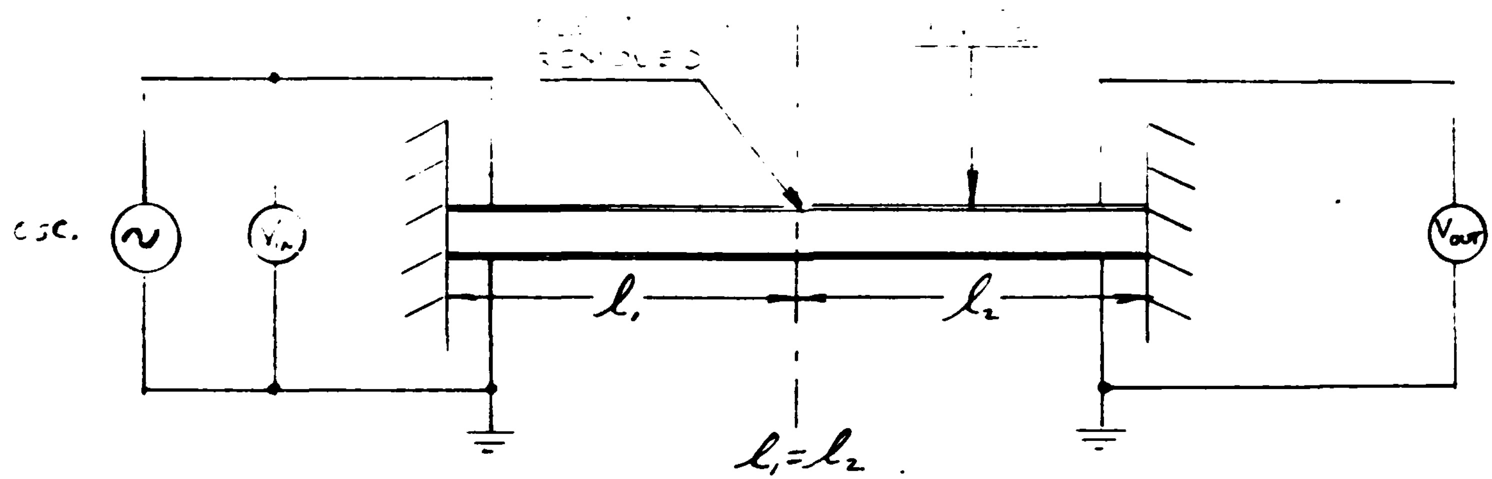

$\ell_1 = \ell_2$

$k_{31}^2 \simeq 2\, V_{OUT}/V_{IN}$

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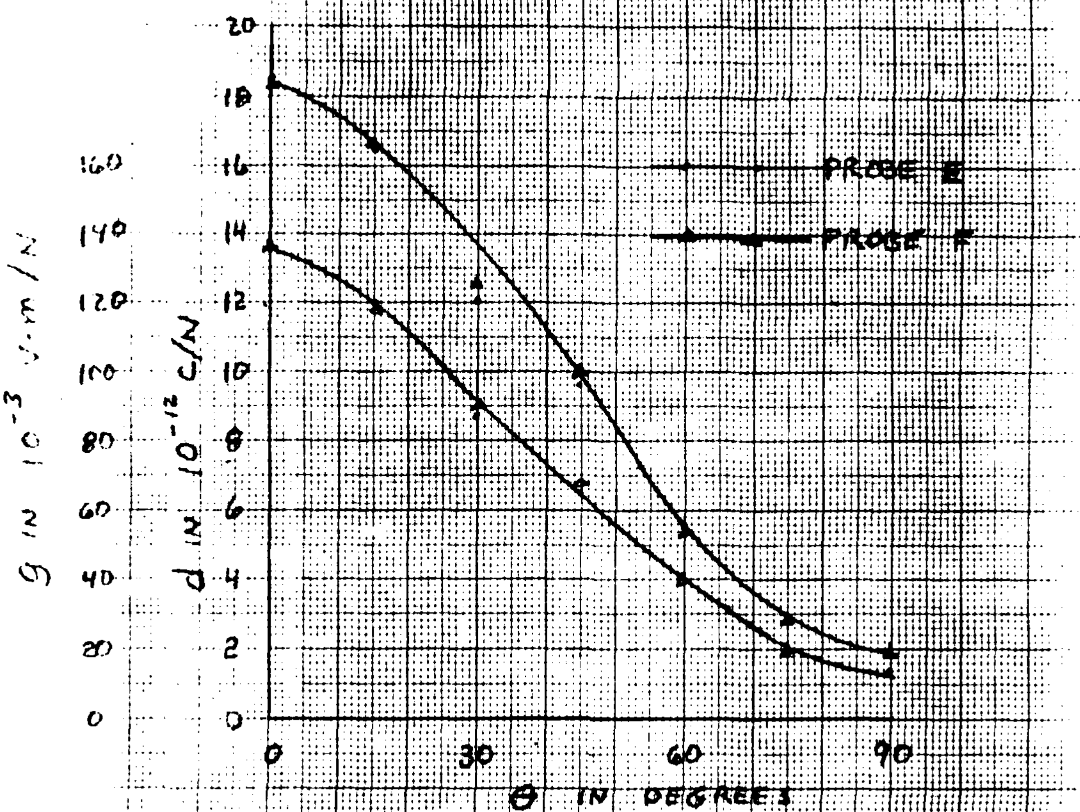

FIG. 4 ANISOTROPY OF PVF_2 AS MEASURED ON d-METER

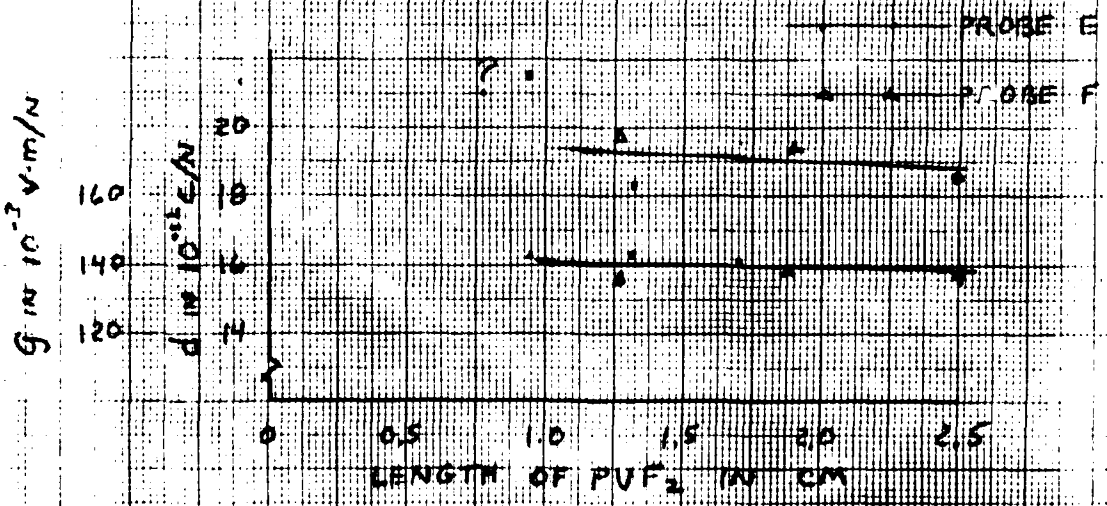

FIG 5 d_{31} + g_{31} VS LENGTH OF PVF_2